### Title of the invention

AN ACTIVE COOLING PANEL OF THERMOSTRUCTURAL COMPOSITE MATERIAL AND METHOD FOR ITS MANUFACTURE

## 5 Background of the invention

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The present invention relates to an active cooling panel of thermostructural composite material.

The term "active cooling panel" is used herein to mean a panel having a cooling fluid passing therethrough for the purpose of taking away the heat received by the panel being exposed to high temperature or high heat flux.

The term "thermostructural composite material" is used mean а composite material having mechanical properties which make it suitable for constituting structural elements and having the ability to conserve these mechanical properties at high temperature. Thermostructural composite materials are typically carbon-carbon (C/C) type composite material comprising a reinforcing structure made of carbon fibers densified by a matrix of carbon, and ceramic matrix composite (CMC) materials comprising a reinforcing structure of refractory fibers (in particular carbon fibers or ceramic fibers) densified by a ceramic matrix.

Applications of the invention lie in particular in constituting the walls of a combustion chamber in an aircraft engine, which walls convey a cooling fluid which may be constituted by the fuel that is injected into the chamber, or walls for the diverging portions of rocket engines which are likewise cooled by fluid, which fluid may be a propellant component injected into the combustion chamber of the rocket engine, or indeed the walls of a plasma confinement chamber in a nuclear fusion reactor. In such applications, the panel acts as a heat exchanger between its face that is exposed to high temperature or high heat flux and the fluid it conveys.

In such heat exchanger walls, the use of active cooling panels made of thermostructural composite material enables the operation of systems including such heat exchangers to be

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extended to higher temperatures and/or enables the lifetime of such systems to be extended. Increasing operating temperature can enable performance to be increased, in particular the efficiency of combustion chambers or nozzles in aviation or space engines, and can also reduce the amount of pollution emitted by aircraft engines.

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Making a part out of thermostructural composite material generally requires a porous fiber preform to be prepared of a shape that is close to the shape of the part that is to be made, with the preform then being densified.

Densification can be performed by a liquid technique or technique. Liquid densification gas consists in impregnating the preform with a liquid that is a precursor of the matrix material, which precursor is generally a resin, and in transforming the precursor, usually by heat treatment. gas technique or chemical vapor infiltration (CVI) consists in placing the preform in an enclosure and in admitting reaction gas into the enclosure, which gas diffuses under determined conditions of pressure and temperature into the pores of the preform and forms a solid deposit therein by one or more of the components of the gas decomposing or reacting Both techniques, using a liquid or CVI are well together. known and they can be combined, for example by performing predensification or consolidation of the preform using a liquid followed by CVI.

Whatever the densification method used, thermostructural composite materials present residual porosity so they are unsuitable for use on their own in forming cooling panels having internal fluid-conveying passages, since the walls of such passages are not leakproof.

Several solutions have been proposed to overcome this difficulty and to enable active cooling by means of a flowing fluid to be combined with the use of porous refractory materials.

A first solution consists in making a panel having a front plate made of graphite on its side that is exposed to

high temperatures, and a rear plate made of metal, in particular steel, with the channels for conveying the cooling fluid being made therein. The two plates are assembled together by brazing, with layers of metal being interposed to match the different coefficients of thermal expansion of steel and of graphite. The presence of solid metal is penalizing in terms of the mass of the cooling panel. In addition, the length of the path along which heat travels through the graphite plate and the metal plate puts a limit on capacity to cool the exposed surface.

Another solution consists in forming passages in a block of thermostructural composite material and in making the walls of the passages leakproof by brazing a metal lining, e.g. made of copper.

Yet another solution consists in making two plates out of thermostructural composite material, one of which plates presents channels machined in its face that is to be assembled with a facing face of the other plate, with assembly being performed by brazing.

The second and third solutions are satisfactory in terms of mass and of shortening heat flow path length, but leakage problems can arise due to the metal lining or the brazing cracking following repeated exposure to very high temperatures and excess stresses induced by the shape of the channels.

#### Object and summary of the invention

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In one of its aspects, the invention seeks to provide an active cooling panel of thermostructural composite material that presents leaktightness which is effective and durable relative to a fluid flowing in internal passages of the panel.

This object is achieved by a panel of the type comprising first and second parts of thermostructural composite material each having an inside face and an opposite outside face, the parts being assembled together by bonding their inside faces together, and channels being formed by indentations formed in the inside face of at least one of the first and second parts,

which panel, according to the invention, further comprises a sealing layer bonded to at least one of the first and second parts and situated at a distance from the assembled-together inside faces thereof.

Such a panel is remarkable in that sealing is achieved not at the interface between the parts, i.e. at the walls of the flow channels, but at a different level of the panel, remote from said interface.

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Thus, the integrity of the sealing layer and its bond with the thermostructural composite material are not affected by excess stresses of the kind that are encountered if the sealing layer is to follow or be subjected to the indentations of the channels at the interface between the parts. In addition, it is then possible to displace the sealing layer further away from the face of the panel that is exposed in operation to high temperatures, thereby reducing the thermomechanical stresses to which the sealing layer is exposed.

In an embodiment of the panel, a sealing layer is situated within at least one of the first and second parts, separating the part into two portions between its inside face and its outside face, the two portions being bonded together by the sealing layer.

In another embodiment, a sealing layer covers at least one of the outside faces of the first and second parts.

Advantageously, the sealing layer is a thin metal layer, for example a metal selected from niobium, nickel, tantalum, molybdenum, tungsten, and rhenium.

When the sealing layer is formed within a part, it is possible to provide for the sealing layer and the portion situated on the outside of the part provided with the sealing layer to project around the periphery of the panel, in particular to facilitate installing a sealing gasket around the periphery of the panel.

Preferably, the channels are formed in the inside face of the part whose outside face constitutes the face of the panel that is to be exposed to high temperatures while the panel is in use.

The panel may be provided with stiffening ribs which project from the outside face of the part situated on the side opposite from the side that is to be exposed to high temperatures while the panel is in use.

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The inside faces of the first and second parts may be bonded together by brazing.

In a variant, the inside faces may be provided with metal coatings that are bonded directly to each other.

In another aspect, the invention seeks to provide a method of manufacturing an active cooling panel as defined above.

This object is achieved by a method of the comprising the steps consisting in providing first and second parts of thermostructural composite material, each having an inside face and an outside face opposite to the inside face, the inside face of at least one of the parts presenting indentations forming channels, and in assembling the first and second parts together by bonding their inside faces together such a manner as to obtain a cooling panel made of thermostructural composite material having circulation channels integrated therein, in which method, according to the invention, at least one of the first and second parts is provided with a sealing layer situated at a distance from the inside face of the part.

In a particular implementation of the method, a sealing layer is integrated within at least one of the first and second parts between its inside face and its outside face.

For this purpose, advantageously, at least one of the first and second parts is made up of two distinct portions, and the portions are assembled together with the sealing layer interposed between them.

In another implementation of the method, the outside face of at least one of the first and second parts is provided with a sealing layer. In either case, the sealing layer may be implemented as a metal foil, e.g. made of a metal selected from niobium, nickel, tantalum, molybdenum, tungsten, and rhenium.

The metal foil may be assembled to the composite material of the first or second part by hot compression, in particular by hot isostatic pressing.

The inside faces of the first and second parts may be assembled together by brazing.

In a variant, it is possible to form at least one metal coating layer on the inside faces of the first and second parts and to assemble said inside faces together by hot compression, in particular by hot isostatic pressing.

Advantageously, prior to assembling together the inside faces of the first and second parts, treatment is performed to reduce the surface porosity of the thermostructural composite material in at least one of said inside faces of the parts.

The treatment for reducing porosity may comprise applying a suspension to the surface of at least one of said inside faces of the part, the suspension comprising a ceramic powder and a ceramic material precursor in solution, the treatment further comprising transforming the precursor into a ceramic material.

The precursor is typically a polymer which is crosslinked and transformed into ceramic by heat treatment.

Optionally, after the precursor has been transformed into ceramic material, chemical vapor deposition or chemical vapor infiltration is performed.

#### Brief description of the drawings

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The invention will be better understood on reading the following description given by way of non-limiting indication and made with reference to the accompanying drawings, in which:

- Figure 1 is a cross-section view of an embodiment of an active cooling panel in accordance with the invention;

- Figure 2 is a fragmentary section view on plane II-II of Figure 1;
- Figure 3 is a section view on plane III-III of Figure 2;
- 5 Figures 4 to 8 show successive steps in implementing a method in accordance with the invention for manufacturing a panel of the type shown in Figure 1; and
  - Figures 9 to 13 are cross-section views of other embodiments of an active cooling panel in accordance with the invention.

# Detailed description of embodiments

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A first embodiment of an active cooling panel 10 is shown in Figures 1 to 3.

The panel 10 comprises two parts 20 and 30 that are generally in the form of rectangular parallelepipeds and that are assembled to each other via their inside faces 21 and 31. In this example, assembly is performed by brazing 12. The part 20 whose outside face 22 opposite from its face 21 defines the front face of the panel that is to be exposed to high temperatures or to intense heat flow is made of a thermostructural composite material. Channels 24 for circulating a cooling fluid are formed by indentations formed in the inside face 21. A plurality of channels 24 parallel to two opposite sides of the panel 10 extend between two manifolds 40, 42 that are internal to the panel 10 and that are situated close to two other opposite sides thereof.

The part 30 comprises two portions 34 and 36 in the form of plates made of thermostructural composite material. The portions 34 and 36 are assembled via facing faces 35, 37 with a sealing layer 38 being interposed between them. The faces of the portions 34 and 36 that are opposite from their faces 35 and 37 define respectively the inside face 31 and the opposite outside face 32 of the part 30. The face 32 constitutes the rear face of the panel 10.

The manifolds 40, 42 are formed by elongate openings or slots formed in the portion 34. The manifolds 40, 42 communicate with the outside of the panel via holes 41, 43 formed through the sealing layer 38 and the portion 36, and provided with metal inserts 44, 46 enabling the panel to be connected with a circuit for circulating fluid and/or with an adjacent panel by means of a connecting coupling.

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In a variant, the channels 24 may each have at least one end opening out into a side end of the part 20. After the cooling panel has been made, the open ends of the channels can then be connected by means of couplings either to a manifold external to the panel, or else to similar channels in an adjacent panel.

The part 20 and the part 30 (portions 34 and 36) are made of a C/C or a CMC thermostructural composite material. For applications at very high temperature, in particular in an oxidizing medium, it is preferred to use CMC, typically comprising composite materials reinforced by silicon carbide (SiC) fibers or carbon fibers with a matrix of SiC or a matrix that has at least an outer phase of SiC. The channels and the manifolds may be made by machining.

Whatever the thermostructural composite material used, it presents residual porosity. The sealing layer 38 makes it possible to prevent any fluid flowing along the channels 24 from leaking to the rear face 32 of the panel 10.

In the example shown in Figures 1 to 3, the part 20 is not provided with a sealing layer. This is acceptable when there is no requirement for a high degree of leaktightness between the channels 24 and the front face 22 of the panel 10. This can apply for an active cooling panel for a combustion chamber wall when the cooling fluid used is a fuel and when a certain amount of leakage into the combustion chamber can be tolerated.

The sealing layer 38 is a metal layer bonded to the faces 35 35, 37 of the portions 34, 36 of the part 30, e.g. a layer of niobium, nickel, tantalum, molybdenum, tungsten, or rhenium.

A method of manufacturing a cooling panel of the kind shown in Figures 1 to 3 is described below with reference to Figures 4 to 8.

The part 20 and the portions or plates 34, 36 of the part 30 are made separately out of thermostructural composite material, in particular C/C or CMC material. The recesses needed for forming the channels 24 and the manifolds are formed by machining the inside face 21 of the part 20 and the portion 34 of the part 30. It should be observed that the part 20 and the portions 34, 36 may be cut out from a single block of thermostructural material prior to machining the locations for the channels and the manifolds.

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The detailed views of Figure 4 show in highly diagrammatic manner the surface porosity of the thermostructural composite material.

Advantageously, treatment is applied to reduce the porosity of the inside face 21 of the part 20 in which the channels 24 are formed, and the face 31 of the portion 34, i.e. those faces that are to be assembled together.

Porosity can be reduced by applying a suspension onto the faces 21 and 31, the suspension containing a solid filler in the form of a ceramic powder and a ceramic precursor in solution, and then transforming the precursor into ceramic material. The precursor may be a polymer which is crosslinked and then transformed into ceramic by heat treatment. By way of example, for the precursor it is possible to use a polycarbosilane (PCS) or a polytitanocarbosilane (PTCS) as a precursor for SiC, which precursor is put into solution in a solvent, e.g. xylene. The ceramic powder contributes to filling in surface pores effectively. It is possible to use an SiC powder, for example.

The liquid composition may be applied using a brush or a spray gun, with the quantity of solvent being selected to make application easy and to encourage penetration of the liquid composition into the surface pores.

After the liquid composition has been applied and has been dried by eliminating the solvent, the precursor polymer is cross-linked and then transformed into ceramic. When using PCS, for example, cross-linking can be performed by raising the temperature to about 350°C, and ceramization by raising the temperature to about 900°C.

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After ceramization, it is optionally possible to shave the surface of the part in order to restore it to its initial shape.

Two detail views in Figure 5 show in highly diagrammatic manner how pores are filled in by the material 51 comprising the ceramization residue and the ceramic powder.

It is also advantageous for pores to be filled in further by forming a deposit of ceramic, e.g. SiC, by chemical vapor infiltration or deposition, thus making it possible to obtain a uniform and continuous coating 52 anchored to the thermostructural composite material.

The ceramic coating 52 obtained by chemical vapor infiltration or deposition (shown in the detailed views of Figure 5) may be formed not only on the inside faces 21 and 31, but also on the other faces on the outside of the part 20, and in particular its outside face 22 and on other surfaces on the outside of the portion 34.

It should be observed that the method of filling in pores by depositing a suspension containing a ceramic powder and a ceramic precursor polymer, and then transforming the precursor into ceramic, followed by shaving and then forming a ceramic coating by chemical vapor infiltration is described in the patent application in the name of the present Applicant and entitled "A method of surface-treating a thermostructural composite material part and its application to brazing thermostructural composite material parts".

The following step of the method consists in interposing a sealing layer between the portions 34 and 36, possibly after machining the faces 35 and 37 of the portions 34 and 36 in order to lay bare the composite material. The sealing layer

is advantageously formed by a metal foil 38 (Figure 6), e.g. made of a metal selected from niobium, nickel, tantalum, molybdenum, tungsten, and rhenium. The thickness of the foil 38 typically lies in the range 0.05 millimeters (mm) to 0.3 mm.

The portions 34 and 36 are bonded together and to the foil 38 by hot compression.

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This can be done using known methods such as the hot isostatic pressing (HIP) assembly method or the method of hot pressing in a press.

Bonding by hot isostatic pressing is performed by placing the elements for assembly against each other in an enclosure while encapsulating the part in а leakproof cover 45 (Figure 7). Temperature and pressure are then raised in substantially uniform manner in the enclosure. achieved by metal from the foil 38 diffusing into the surface faces 35, pores of the 37. The leakproof cover 45 encapsulating the parts is constituted, for example, by a metal film such as a film of niobium, or indeed of nickel, of iron, or of an alloy thereof. Tooling elements such as plates of graphite 46, 47 may be interposed between the metal film and the outside surfaces of the portions 34, 36 in order to prevent the metal of the film 45 becoming embedded in said surfaces due to the hot isostatic pressing when the presence of said metal on said surfaces is undesirable. This may apply in particular to the face 31, depending in particular on the method used subsequently for bonding it to the face 21 of the part 20.

Bonding by pressing in a press consists in raising the temperature of the elements to be assembled together and in pressing them against one another by exerting pressure on the faces 31 and 32 in a press.

The pressure used for hot compression bonding lies, for example, in the range 80 megapascals (MPa) to 120 MPa. The temperature is a function of the nature of the metal sealing layer used for bonding the parts together. It is

substantially lower than the melting temperature of the metal of said metal layer, generally lying in the range 60% to 80% of said melting temperature.

When the metal sealing layer is made of niobium, the temperature is selected more particularly to lie in the range 900°C to 1200°C both for bonding by hot isostatic pressing and for bonding by pressing in a press.

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Once the part 30 has been made, it is assembled to the part 20, e.g. by brazing. For this purpose, a layer of brazing 48 is interposed between the reduced-porosity faces 21 and 31 (Figure 8).

Brazing together parts made of thermostructural composite material is, in itself, known. For example, it is possible to use a brazing material based on silicon of the type described in the French patent applications published under the Nos. 2 748 471 and 2 749 787. Other brazing compositions can be used, in particular compositions based on silicon or on titanium such as those sold under the name TiCuSil® by Wesgo Metals, a division of the US supplier Morgan Advanced Ceramics.

In a variant, the parts 20 and 30 can be bonded together by hot compression.

For this purpose, the surfaces 21 and 31 are initially provided with metal coatings which, in addition to providing bonding by hot compression, can also perform a sealing function.

By way of example, each face 21, 31 is provided with a first layer of a metal that advantageously performs a barrier function against chemical reaction with the underlying material and/or a matching function, and a second metal layer having the ability to bond by hot compression.

The second layer may be a metal selected from nickel, copper, iron, or an alloy of at least one of them. Nickel (Ni) or a nickel alloy present the advantages of good thermal conductivity, good ability to bond by hot compression, and a

high melting temperature avoiding passage into the liquid state during bonding by hot compression.

The first layer may be made of a metal selected from molybdenum, rhenium, tungsten, and tantalum. thermostructural composite material has an SiC matrix fiber reinforcement of carbon or of SiC, and/or when a coating of SiC has previously been formed thereon, rhenium presents the advantage of not reacting with SiC. It also presents good conductivity and it has a high melting temperature ensuring that it does not pass to the liquid state during subsequent bonding under hot compression. Furthermore, rhenium has a coefficient of expansion that is intermediate between those of and Ni and therefore also constitutes a mechanical matching layer when the second metal layer is constituted at least in part by Ni.

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The first and second metal layers are deposited in succession. It is possible to use conventional deposition methods of the physical vapor deposition type or the plasma sputtering type.

Prior to bonding the parts together by hot compression, a metal foil may be interposed between the facing inside faces of the parts, which metal foil is preferably made of the same material as the second metal layer of the metal coating formed on the inside surfaces 21, 31.

The parts 20 and 30 are bonded together by hot compression, possibly after inserting a metal foil.

It is possible to use the hot isostatic pressing assembly method or the method of pressing in a press as described above.

When the parts 20 and 30 are bonded together by hot compression, it is possible to make said bond simultaneously with the bond between the portions 34, 36 and the sealing layer 38, after forming the metal coatings on the inside faces 21 and 31.

Figures 9 to 13 illustrate various other embodiments of an active cooling panel in accordance with the invention.

Thus, the panel of Figure 9 differs from that of Figures 1 to 3 in that the portion 36 of the part 30 and the sealing layer 38 project around the periphery of the panel.

The panel can then be housed in a frame 54 comprising a base 55 from which there projects a rim 56. A sealing gasket 58 is disposed in the space defined by the base 55, the periphery of the panel 30 in the vicinity of the part 20 and the portion 34, the projecting portion 36 and layer 38, and the rim 56. The gasket 58 serves to contain cooling fluid leaks around the periphery of the panel.

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The panel of Figure 10 differs from that of Figures 1 to 3 in that the part 30 is provided with stiffeners 60. These are in the form of stiffening ribs projecting from the outside face 32 of the portion 36 of the part 30.

The ribs 60 may be made integrally with the portion 36.

The ribs 60 give the panel greater ability to withstand the forces to which it is subjected, preventing deformation which might damage the bonds between the portions 34, 36 of the part 30 and between the parts 20 and 30.

The panel of Figure 11 differs from that of Figures 1 to 3 in that not only the part 30, but also the part 20 is provided with a sealing layer 62 integrated within the part 20 and at a distance from the interface between the parts 20 and 30.

25 The layer 62 may be of the same kind and may be put into place in the same manner as the sealing layer 38, in which case the part 30 is likewise made by assembling together two distinct portions with the layer 62 being interposed between them.

30 The panel of Figure 12 differs from that of Figures 1 to 3 in that the part 30 is a single piece of thermostructural composite material and the sealing layer 64 is disposed on the outside face 32 of the part 30 instead of being disposed within it.

The layer 64 may be of the same kind as the sealing layer 38 and it may likewise be assembled to the part 30 by hot compression.

As shown in Figure 13, the part 20 of a panel of the kind shown in Figure 12 may also be provided with a sealing layer 66 assembled to its outside face 22.

The panels of Figures 12 and 13 are easier to make than those of the other panels. However integrating the sealing layer within a part, between two portions of thermostructural composite material, enables said sealing layer to be protected against oxidation by the presence of the composite material. In addition, placing the sealing layer on the outside face of a part can make it necessary for the sealing layer to be shaped so as to take account of the possible presence of stiffeners or interfaces with the outside of the panel.

Naturally, a single panel may be provided with a sealing layer based on an outside face of one of the two parts of the panel, and with a sealing layer disposed within the other part.

It is also possible to place the panels of the embodiments shown in Figures 10 and 11 in a frame, as shown for the embodiment of Figure 9.

## Example

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A part 20 and portions 34, 36 of the kind shown for the embodiment of Figures 1 to 3 have been made out of C/SiC thermostructural composite material, with the channels and the manifolds being formed by machining.

The porosity of the inside surfaces 21, 31 was reduced by brushing thereon a composition containing an SiC powder of mean grain size equal to about 9 microns ( $\mu m$ ) in a solution of PCS in xylene. After drying in air, the PCS was cross-linked at about 350°C and then transformed into SiC by raising the temperature to about 900°C. A thin coating of SiC having thickness equal to about 100  $\mu m$  was then deposited by chemical vapor infiltration, said coating then being formed over the

entire outside surface of the part 20 and the portion 34, and not only on the inside faces 21 and 31. In combination with the residue of ceramizing the PCS in association with the SiC powder, the SiC coating contributes to effective reduction of porosity.

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The faces 35, 37 of the portions 34, 36 were then machined in order to lay bare the composite material so as to present open pores, encouraging mechanical bonding with the foil subsequently put into place between these faces. A 0.1 mm thick niobium foil was interposed between the faces 35 and 37, and assembly was then performed by hot isostatic pressing. For this purpose, the elements 34, 38, and 37 were encapsulated in a 0.5 mm thick niobium foil with plates of graphite being interposed between the outside surfaces of the elements to be assembled together and the niobium foil.

Hot isostatic pressing was performed at a pressure of about 90 MPa and at a temperature of about 1000°C.

The part 30 as obtained in this way was assembled to the part 20 by brazing using a silicon-based brazing composition.